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COMMITTEE OF EXPERTS ON THE
TRANSPORT OF DANGEROUS GOODS

Sub-Committee of Experts on the
Transport of Dangerous Goods

REPORT OF THE SUB-COMMITTEE OF EXPERTS ON ITS NINTH SESSION
(Geneva, 4 - 15 July 1994)

Addendum 3

Annex 3

REPORT OF THE WORKING GROUP ON THE RATIONALIZATION OF THE MANUAL OF TESTS AND CRITERIA

Part II: Parts II and III of the Manual

Introduction

1. A working group was established from 11 to 14 July 1994 to develop further Parts II and III of the draft rationalized Manual of Tests and Criteria proposed by the expert from the United Kingdom, in document ST/SG/AC.10/C.3/R.475, which was prepared on the basis of discussions at:

an inter-sessional working group held at BAM in March 1993 (see document ST/SG/AC.10/C.3/R.366);

the seventh session of the Sub-Committee (see document ST/SG/AC.10/C.3/14, paragraphs 5 to 19 and annex 1); and

the eighth session of the Sub-Committee (see document ST/SG/AC.10/C.3/16, paragraphs 6 to 14, 25 to 34 and annex 1).

The terms of reference of the group were limited by the Sub-Committee to an editorial review and rationalization of the General Introduction, Parts II and III and the relevant appendices to the manual. Technical amendments which had been proposed in documents could also be considered provided they did not change

the classification criteria. The working group was also requested to consider documents concerning organic peroxides and self-reactive substances. It was chaired by Mr. Groothuizen and included experts from: France, Germany, Japan, Netherlands, Norway, Sweden, United Kingdom, United States and from the European Chemical Industry Council (CEFIC) and European Fertilizers Manufacturers Association (EFMA).

2. The following documents were considered by the working group:

Relating to oxidizing substances:

-/C.3/R.480/Rev.1	Tests for solid and liquid oxidizers (J);
-/C.3/R.496	Liquid oxidizing substances (S);
-/C.3/R.498	Solid oxidizing substances (N);
-/C.3/R.528	New and revised test methods for Parts II and III (Secretariat);
-/C.3/R.534	Liquid oxidizing substances and solutions (N);
INF. 25	Amended criteria for the revised solid oxidizer test (CEFIC); and
INF. 26	Test data from the revised solid oxidizer test (CEFIC).

Relating to the test manual:

-/C.3/R.385/Rev.1	Test methodology for self-heating substances;
-/C.3/R.441	Substances with low explosion hazards (G);
-/C.3/R.474	General Introduction and Part I of the draft rationalized test manual (UK);
-/C.3/R.475	Parts II and III and Appendixes of the Manual (UK);
-/C.3/R.476	Consequential amendments to the Recommendations (UK);
-/C.3/R.477	Revised Test series H (NL);
-/C.3/R.528	New and revised test methods for Parts II and III (Secretariat);
-/C.3/R.557	Test H.4 Heat accumulation storage test (G);
-/C.3/R.558	Test A.1 BAM 50/60 tube test (G).

Relating to organic peroxides and self-reactive substances:

-/C.3/R.545	Harmonisation of temperature control requirements (USA);
-/C.3/R.571	Division 5.2: 11.3.8 General Packing Requirements (CEFIC);
-/C.3/R.572	Test method for emergency vent sizing;
-/C.3/R.573	Amendments to Table 14.1 (CEFIC);
-/C.3/R.575	Desensitization of organic peroxides (CEFIC);
-/C.3/R.576	Amendments to Tables 11.3 and 11.4 (CEFIC);
-/C.3/R.577	Division 5.2: Simplification of packing methods (CEFIC);
-/C.3/R.579	IBCs for organic peroxides (CEFIC);
-/C.3/R.581	Tables 11.3 and 11.4 (CEFIC); and
-/C.3/R.586	Additional packagings for organic peroxides (USA).

Procedure

3. At the request of the German delegation, the oxidizer tests were discussed first. For each test:

discussion, first centred on the principle;

where complete agreement was not possible, a vote was held to indicate the balance of opinion; and

test prescriptions were amended so that, if they were adopted by the Sub-Committee, no further changes would be necessary.

The proposed changes to the annex to document -/C.3/R.528 and annex 1 to document -/C.3/R.496 are given in annexes A and B respectively.

4. As the latest version of the draft rationalized manual was available as official documents, -/C.3/R.474 and -/C.3/R.475, it was agreed that the working group should produce a narrative report and annexes giving the proposed text amendments to these documents and the Recommendations. Annexes C and D give the changes to documents -/C.3/R.474 and -/C.3/R.475 respectively and annex E gives the consequential amendments to the Recommendations.

5. Most of the text in square brackets was agreed, as was the text proposed for deletion. Minor changes are not discussed in this report but are given in the annexes. This report concentrates on the contentious issues, on the inclusion of new "UN" test methods and on areas where it was considered that additional, clarifying text was necessary.

OXIDIZERS

Solid oxidizer tests

6. After introducing the documents concerning solid oxidizer tests, there was clarification of areas of concern, introduction of new test results (INF.26: on laboratory chemicals which usually have a smaller particle size than industrial chemicals) and a general discussion. It emerged that there was no complete agreement on the way forward. Apart from the United States, experts from all voting countries, together with representatives of industry, accepted that the current test, as given in Chapter 11 of the Recommendations, has serious deficiencies. Experts from Japan and the United States were in favour of improving the test in the way suggested by Japan in document -/C.3/R.480/Rev.1, whilst those from France, Germany, Netherlands, Norway, Sweden and the United Kingdom, and industry representatives, favoured improvement of the test in the way proposed in document -/C.3/R.528 but with criteria as amended in document INF. 25. It was recognised that the particle size of the oxidizer greatly influences its behaviour in the test and it was agreed that the particle size of the substance tested should be stated in the test report. Amendments were made

to the annex of document -/C.3/R.528 but it was agreed that the Sub-Committee should decide whether it should be adopted.

7. The expert from the United States opposed replacing the current test with the new (French) test (document -/C.3/R.528) because the results shown in document INF.25 indicate that the new test will cause a large number of oxidizers to change their current packing group assignment. He considered that if the revised prescription and criteria were adopted then the current Packing Group assignments should be altered in accordance with the test criteria. Other experts considered that this would only be necessary on safety grounds (see paragraph 11.2.1.2 of the Recommendations). A third alternative was to allow a range of Packing Groups depending on the particle size etc of the substance as is the current position with the N.O.S. entries. After further discussions, it was agreed that the best way forward was to adjust the Packing Group I/II criterion in document INF.25 to minimise the effect on the current packing group assignments. It was therefore agreed that, apart from the expert from Japan, the revised solid oxidizer test description, proposed in document -/C.3/R.528, could be accepted. However, it was agreed that the revised Packing Group I/II criterion should be in square brackets until the Committee meeting so that countries had a final opportunity to check the existing data and perform further tests if necessary. The current data, assessed according to the revised criteria, is given in annex A.

Liquid oxidizer tests

8. The Japanese and Swedish documents, -/C.3/R.480/Rev.1 and -/C.3/R.496 respectively, on liquid oxidizer tests were briefly introduced. Only the expert from Japan was prepared to recommend adoption of the Japanese test at this time. It was made clear that the results given in document -/C.3/R.496 were a mixture of results from previous versions of the test and the final version of the test, with an improved procedure. The results from the final version of the test were much more reproducible. The Norwegian results (see -/C.3/R.534) were obtained using the latest version of the test. Experts from five voting countries (France, Germany, Netherlands, Sweden and the United Kingdom) were in favour of adopting the Swedish test, two were against (Japan and United States) and one abstained (Norway). Industry representatives were divided on whether it was appropriate to adopt the Swedish test in this biennium but were not against the test in principle. The experts from Norway and the United States considered that more work should be done in developing the Japanese test as it was simpler to perform.

TEST MANUAL

General Introduction

9. Table 1.3 was amended to reflect the decisions with regard to the recommended tests for Part II of the manual.

Introduction to Part II

10. As proposed in paragraph 3 of document -/C.3/R.528, it was agreed that it was no longer necessary to perform cavitated detonability tests and figure 20.1(b) was amended accordingly.

Test Series A

11. In paragraph 2 of document -/C.3/R.528, it was proposed that the A.1 to A.4 test methods should be deleted. The working group on the General Introduction and Part I of the manual had agreed a new UN Gap test. It was agreed that the United States Gap test (test A.3) and the French Gap test (test A.4) could be deleted and replaced by the UN Gap test, renumbered test A.5, and modified to include criteria suitable for self-reactive substances and organic peroxides (see annex D for the new test prescription). The UN Gap test proposed by the Netherlands, given as test A.5 in document -/C.3/R.475, was renamed the UN Detonation test, renumbered A.6 and remains the recommended test.

12. It was agreed that the screening procedure for detonability, modified to reflect the subsequent decision not to accept the UN Pressure Vessel test, was suitable for organic peroxides but not for self-reactive substances.

13. All the experts, apart from Germany, were in favour of deleting the requirement for performance of cavitated detonability tests as proposed in paragraph 3 of document -/C.3/R.528. All the references in Test Series A to the performance of cavitated tests on liquids were deleted.

14. The amendments to the BAM 50/60 tube test, proposed in document -/C.3/R.558, were accepted apart from those in paragraphs 5 and 7 relating to performance of a cavitated version of the test.

Test Series B

15. Only minor amendments were made to Test Series B.

Test Series C

16. The proposal in paragraph 4 of document -/C.3/R.528, to use the Time/Pressure test only for solids and the Deflagration test only for liquids, was not accepted. A footnote on problems with testing liquids in the Time/Pressure test was inserted along the lines agreed for the Series 1 and 2 versions of the tests.

Test Series D

17. Only minor editorial amendments were made.

Test Series E

18. The UN Pressure Vessel test was deleted and the combinations of tests to be used for self-reactive substances and organic peroxides agreed, as proposed in paragraphs 5 and 6 of document -/C.3/R.528.

19. The Koenen test was amended as agreed for the Series 1 and 2 versions and the Dutch Pressure Vessel test and United States Pressure Vessel test were edited in the same manner.

20. It was agreed, as proposed in paragraph 5 of document -/C.3/R.528, that work should continue on the development of an improved version of the Thermal Explosion Vessel Test.

Test Series F

21. It was agreed that the Modified Trauzl test should be the recommended test but it was recognised that this could cause problems in the future as it used lead and could only be used for low energy materials. There was some discussion on whether the Ballistic Mortar Mk.IIId should be the recommended test. Only minor amendments were made to the test prescriptions.

Test Series G

22. Only minor editorial amendments were made.

Test Series H

23. The text given in document -/C.3/R.477 was discussed in detail. However, a considerable number of changes are required and it was agreed that these should be prepared by the expert from the United Kingdom and circulated to the countries of test origin for comment. The expert from the United Kingdom will then provide text amendments to document -/C.3/R.475 for consideration by a working group at the Committee meeting.

Introduction to Part III

24. No changes were made and the reserved sections for Classes 2, 6, 7 and 8 were maintained.

Class 3

25. Only minor editorial amendments were made.

Class 4

26. The test and criteria for the test method for self-heating substances was revised as proposed in document -/C.3/R.385/Rev.1 and agreed by the Sub-Committee at this session with the amendment, agreed with representative from CEFIC, that a 60°C temperature rise be taken as a positive result in each version of the test. This sets the criteria for a positive for test at 100°C and 120°C to that for the current test at 140°C. Test results are required for tests N.1, N.2, N.4 and N.5. A representative from CEFIC provided results for test N.3.

27. Document -/C.3/R.441 was discussed in general terms. It was agreed that the document should be deferred to the next biennium. Some experts considered that the best way to proceed would be to develop a few specific N.O.S. entries with criteria linked to the Class 1 acceptance and assignment procedures and the text in paragraphs 4.4.4 and 4.4.5 of the Recommendations. It was recognised that there were problems with the air transport of substances related to self-reactive substances and the road and rail transport of desensitized explosives.

Class 5: Division 5.1

28. Paragraph 34.3.1 was amended to reflect the importance of particle size (see paragraph 6 of this report).

Class 9

29. Only minor editorial amendments were made.

Appendices

30. The national test contact for Sweden needs to be inserted in appendix 4 if the Swedish liquid oxidizer test is accepted.

RECOMMENDATIONS

31. The consequential amendments to Chapters 5, 11 and 14 of the Recommendations, proposed in document -/C.3/R.476, were agreed.

ORGANIC PEROXIDES AND SELF-REACTIVE SUBSTANCES

Harmonisation of temperature control requirements

32. It was agreed that the current temperature control requirements were too complicated and needed rationalizing. However, as major consequential amendments would be required, it was agreed that it would be best to do this in a future meeting. The main concern with transporting type G organic peroxides and self-reactive substances was that they may be transported in tanks or IBCs without

being subjected to adequate safety requirements. It was considered that restricting exemption to 440 kg package, as has just been agreed for Division 4.2 self-heating substances, may offer a solution. The proposals in document -/C.3/R.545 and a similar proposal, in annex 2 of document -/C.3/R.477, to harmonise the temperature control requirements were withdrawn for further consideration.

Amendments to table 14.1

33. The proposal (document -/C.3/573) to classify a mixture of monomer with a small amount of peroxide initiator as a self-reactive liquid of Division 4.1 was agreed with minor amendments.

Desensitization of organic peroxides

34. It was agreed that there was a gap in the definition of diluents type A and B for organic peroxides which unnecessarily prevented use of certain diluents. However, acceptance of the CEFIC proposal in document -/C.3/R.575 would involve a considerable number of consequential amendments which not been addressed. Instead of their proposal, the definition of diluent type B was changed in order to fill the gap (see annex E).

Amendments to tables 11.3

35. CEFIC documents -/C.3/R.576 and -/C.3/R.581 were discussed on the basis of additional test results provided to the working group. Some alterations to the proposed classifications were discussed but, since all the required data was not available, CEFIC decided to withdraw their documents for further consideration and to provide one new document for the Committee with a complete list of products and proposed classifications, a summary of the supporting test data and the proposed consequential amendments. These could be discusssed by a working group on Parts II and III of the test manual.

Test method for emergency vent sizing

36. All agreed that there were considerable advantages in having an example test method for sizing the emergency relief systems on tanks as required in paragraph 12.550 of the Recommendations. It was considered inappropriate to insert the full test method in either the test manual or the Recommendations at this time. However, it was considered useful to include a reference in Chapter 11 of the Recommendations (see annex E) to the CEFIC document. The working group understood from the Secretary that this was possible.

IBCs and packagings

37. Documents -/C.3/R.571, -/C.3/R.577, -/C.3/R.579 and -/C.3/R.586 were briefly discussed. It was agreed to prepare revised documents, based on the discussions, which would give full details and, where appropriate, consequential amendments.

Actions requested of the Sub-Committee

38. The Sub-Committee is invited to note this report and include it as an annex to the official report as a record of the principles underlying the agreed amendments to documents -C.3/R.474, -/C.3/R.475, -/C.3/R.496, -/C.3/R.528 and the Recommendations, and of the outstanding actions.

39. All delegations are requested to provide further test results, particularly where none or few are currently given.

40. The working group believes that it will be necessary to have a formal working group at the next meeting of the Committee on:

Test Series H of Part II of the manual;

the oxidizer tests; and

revised documents relating to organic peroxides and self-reactive substances that are referred by the Committee.

If the Sub-Committee agrees to this, the working group is confident that the work on rationalization of the manual can be completed in this biennium. The Sub-Committee is invited to set up a two day working group, starting at 10.00 a.m. on 28 November, to complete the work.

41. The Sub-Commmittee is invited to adopt the revised version of the solid oxidizer test, given in document -/C.3/R.528, with the revised criteria given in annex A to this report and subject to the Packing Group I/II criterion remaining in square brackets until the Committee meeting.

42. The Sub-Committee is invited to carry document -/C.3/R.441 forward to the next biennium and to give consideration to developing specific N.O.S. entries, with classification principles linked to the Class 1 acceptance and assignment procedures, for desensitized explosives and substances related to self-reactive substances in the next biennium.

43. The Sub-Committee is invited to carry the following documents forward to the Committee meeting:

-/C.3/R.474	General Introduction and Part I of the draft rationalized test manual (UK);
-/C.3/R.475	Parts II and III and Appendixes of the Manual (UK);
-/C.3/R.480/Rev.1	Tests for solid and liquid oxidizers (J);
-/C.3/R.496	Liquid oxidizing substances (S);
-/C.3/R.534	Liquid oxidizing substances and solutions (N);

The Sub-Committee is advised that the following documents have been discussed to a conclusion:

-/C.3/R.385/Rev.1	Test methodology for self-heating substances;
-/C.3/R.476	Consequential amendments to the Recommendations (UK);
-/C.3/R.477	Revised Test series H (NL);
-/C.3/R.498	Solid oxidizing substances (N);
-/C.3/R.528	New and revised test methods for Parts II and III (Secretariat);
-/C.3/R.545	Harmonisation of temperature control requirements (USA);
-/C.3/R.557	Test H.4 Heat accumulation storage test (G);
-/C.3/R.558	Test A.1 BAM 50/60 tube test (G).
-/C.3/R.571	Division 5.2: 11.3.8 General Packing Requirements (CEFIC);
-/C.3/R.572	Test method for emergency vent sizing;
-/C.3/R.573	Amendments to Table 14.1 (CEFIC);
-/C.3/R.575	Desensitization of organic peroxides (CEFIC);
-/C.3/R.576	Amendments to Tables 11.3 and 11.4 (CEFIC);
-/C.3/R.577	Division 5.2: Simplification of packing methods (CEFIC);
-/C.3/R.579	IBCs for organic peroxides (CEFIC);
-/C.3/R.581	Tables 11.3 and 11.4 (CEFIC); and
-/C.3/R.586	Additional packagings for organic peroxides (USA).

Annex A

AMENDMENTS TO THE ANNEX TO DOCUMENT ST/SG/AC.10/C.3/R.528

Delete all square brackets or, where indicated, the text in square brackets.

In paragraph 34.4.1.1, change "1:3" to "3:7", "1:2" to "2:3" and "1:1" to "[3:2]".

In paragraph 34.4.1.2.2, delete the text in square brackets and add a footnote after "cellulose" to read:

"*/ available from the national contact for test details in France (see appendix 4)."

In paragraph 34.4.1.2.4, amend "heat transfer coefficient" to read "thermal conductivity".

In paragraph 34.4.1.2.6, amend "1%" to read "10%".

In paragraph 34.4.1.3.1, change "1:3" to "3:7", "1:2" to "2:3" and "1:1" to "[3:2]".

In paragraph 34.4.1.3.3, insert after "incandescence", "glowing combustion" and insert, after "Intermittent reaction", ", such as sparking or sputtering,".

In paragraph 34.4.1.4.2; under Packing Group I, change "1:1" to "[3:2]"; under Packing Group II, change "1:2" to "2:3"; under Packing Group III, change "1:3" to "3:7"; and under Not Division 5.1, change "1:3" to "3:7".

In paragraph 34.4.1.5 after the Note, replace the current table with:

Substance	Mean burning times (s)		Results
	4:1	1:1	
Ammonium dichromate	55	189	PG III a/
Ammonium nitrate (crystalline)	161	74	PG III
Calcium nitrate (unhydrous)	10	25	PG II d/
Calcium nitrate (tetra hydrate)	268	142	Not 5.1
Ceric ammonium nitrate	10	36	PG II b/
Chromium trioxide	3	33	PG I c/
Cobalt nitrate (hexahydrate)	205	390	Not 5.1
Nickel nitrate	101	221	Not 5.1 d/
Potassium nitrite	8	15	PG II
Potassium perchlorate	9	33	PG II
Potassium permanganate	17	51	PG II
Sodium chlorate	5	13	PG II
Sodium nitrite	15	22	PG II d/
Sodium nitrate	56	39	PG II d/
Strontium nitrate (anhydrous)	107	237	Not 5.1 d/
Mixtures of reference substance to fuel			
3:7 Potassium bromate/cellulose	100		
2:3 Potassium bromate/cellulose	54		
[3:2] Potassium bromate/cellulose	4		

[a/ Currently PG. II but borderline
b/ Not current classified
c/ Currently PG. II but borderline
d/ Currently PG. III]

In Figure 34.4.1.1, after "(A) Base ...", insert "(70 mm diameter)".

Annex B

AMENDMENTS TO ANNEX 1 OF DOCUMENT ST/SG/AC.10/C.3/R.496

Delete all square brackets or, where indicated, the text in square brackets.

Amend the footnote to "determined*/" to read:
"*/ In some cases, substances may generate a pressure rise (too high or too low), caused by chemical reactions not characterising the oxidizing properties of the substance. In these cases, it may be necessary to repeat the test with an inert substance, e.g. diatomite (kieselguhr), in place of the cellulose in order to determine whether it is a false effect."

In 34.4.2.2.1, replace "BSP", by "British Standard Pipe (BSP)".

In 34.4.2.2.2, insert "inert" before "seal".

In 34.4.2.2.5, replace the first sentence by:
"Dried, fibrous cellulose */ with a fibre length between 50 and 250 μ m and a mean diameter of 25 μm, is used as the combustible material."

Insert a new footnote to read:
" */ available from the national contact for test details in Sweden (see appendix 4)."

Insert a new paragraph 34.4.2.2.2.6 to read:
"34.4.2.2.2.6 50% perchloric acid, 40% aqueous sodium chlorate solution and 65% aqueous nitric acid are required as reference substances."

In 34.4.3.2.3.2, append to the sentence beginning "Five trials .." text to read:
"and each of the reference substances".

Annex C

AMENDMENTS TO THE ANNEX OF DOCUMENT ST/SG/AC.10/C.3/R.474

Amend table 1.3 to read:

"Table 1.3: RECOMMENDED TESTS FOR SELF-REACTIVE SUBSTANCES AND ORGANIC PEROXIDES

Test series	Test code	Test name
A	A.6	UN detonation test
B	B.1	Detonation test in package
C	C.1	Time/pressure test
C	C.2	Deflagration test
D	D.1	Deflagration test in the package
E	E.1	Koenen test
E	E.2	Dutch pressure vessel test
F	F.4	Modified Trauzl test
G	G.1	Thermal explosion test in package
H	H.1	United States SADT test (for packages)
H	H.2	Adiabatic storage test (for packages, IBCs and tanks)
H	H.4	Heat Accumulation Storage Test (for packages, IBCs and small tanks)

Annex D

AMENDMENTS TO THE ANNEX OF DOCUMENT ST/SG/AC.10/C.3/R.475

Delete all square brackets or, where indicated, the text in square brackets and delete all editorial and technical notes.

Section 20

In Figure 20.1(b), delete Boxes 14 and 15.

In Figure 20.2, in 5.3 and 5.7 amend "t_1" to read "time to reaction", "t_2" to read "duration of reaction", delete 7.3 and renumber all subsequent sub-sections.

Section 21

In 21.1.1, amend "boxes 1, 14, and 15" to read "box 1".

In Table 21.1, delete the entries A.3 and A.4, for the entry A.5, change the reference to "21.4.3" and delete the "*/", and add a new entry to read:

"A.6 UN Detonation test */ 21.4.4".

In paragraph 21.2.2, replace "A" with "For organic peroxides a", amend "E.1 and test E.5" to read:

"E.2 and either test E.1 or E.3" and delete the last sentence and the footnote.

Delete paragraph 21.2.3 and the footnote.

In 21.4.1.1, amend the final sentence to read "... question in box 1 of figure 20.1.".

In 21.4.1.2, replace the first sentence by:

"A seamless drawn steel tube of 500 mm length, 60 mm external diameter and 5 mm wall-thickness (e.g. according to DIN 2448) should be used, made from steel St 37.0 with tensile strength of 350 to 480 $N.mm^{-2}$ (e.g. according to DIN 1629). The tube is closed by a malleable cast iron screwing cap or by an appropiate plastic cap, put over the open end of the tube.".

In 21.4.1.2, replace "St. 37" by "St. 37.0", and append:

"When a fresh batch of steel tubes is used, a calibration test using water should be performed to determine the blank fragmentation length. If the fragmented length exceeds 14 cm, the criterion in 21.4.1.4.2 should be recalculated by adding 7 cm to this experimentally determined value. The criteria should be adjusted accordingly."

Delete paragraph 21.4.1.3.1, but not the sub-paragraphs.

Renumber "21.4.1.3.1.1" to "21.4.1.3.1".

Delete paragraph 21.4.1.3.3.

Delete paragraph 21.4.1.5.1, but not the sub-paragraphs (renumbered as appropriate).

In Table 21.4.1.5, delete the reference to and footnote b/, add the entries listed in paragraph 6 of document -/C.3/R.558 with the amendments: replace the references to footnotes c/ and d/ by b/ and c/ respectively.

Delete paragraph 21.4.1.5.2.

In Figure 21.4.1.1 delete "made from steel ....St. [37]".

In 21.4.2.1, amend the final sentence to read: "... question in box 1 of figure 20.1.".

In 21.4.2.2.1, insert before material "e.g.".

Delete paragraph 21.4.2.3.1.

Renumber paragraph 21.4.2.3.1.1 to 21.4.2.3.1 and paragraph 21.4.2.3.1.2 to 21.4.2.3.2.

Delete paragraph 21.4.2.3.2.

Delete "21.4.2.5.1. Results ... figure 20.1)".

Delete paragraph 21.4.2.5.2.

Delete sections 21.4.3 and 21.4.4.

Insert a new section 21.4.3 containing the Series 1 version of the UN Gap test as follows:

"21.4.3 ***Test A.5: UN gap test***

21.4.3.1 *Introduction*

This test is used to measure the ability of a substance to propagate a detonation by subjecting it to a detonating booster charge under confinement in a steel tube. It may be used to answer the question in box 1 of figure 20.1.

21.4.3.2 *Apparatus and materials*

The apparatus is shown in figure 21.4.3.1. The test sample is contained in cold-drawn, seamless, carbon steel tube with an external diameter of 48 ± 2 mm, a wall thickness of 4.0 ± 0.1 mm and a length of 400 ± 5 mm. If the test substance may react with the steel, the inside of the tube may be coated with fluorocarbon resin. The bottom of the tube is closed with two layers of 0.08 mm thick polythene sheet pulled tightly (so that it plastically deforms) over the bottom of the tube and held in place with rubber bands and insulating tape. For samples which affect polythene, polytetrafluoroethylene sheet can be used. The booster

charge consists of 160 g RDX/wax (95/5) or PETN / TNT (50/50), 50 ± 1 mm in diameter with a density of 1600 ± 50 kg/m^3 giving a length of about 50 mm. The RDX/wax charge may be pressed in one or more pieces, as long as the total charge is within the specifications, and the PETN / TNT charge is cast. A mild steel witness plate, 150 ± 10 mm square and 3.2 ± 0.2 mm thick, is mounted at the upper end of the steel tube and separated from it by spacers 1.6 ± 0.2 mm thick.

21.4.3.3 *Procedure*

21.4.3.3.1 The sample is loaded to the top of the steel tube. Solid samples are loaded to the density attained by tapping the tube until further settling becomes imperceptible. The sample mass is determined and, if solid, the apparent density calculated using the measured internal volume of the tube. The density should be as close as possible to the shipping density.

21.4.3.3.2 The tube is placed in a vertical position and the booster charge is placed in direct contact with the sheet which seals the bottom of the tube. The detonator is fixed in place against the booster charge and initiated. Two tests should be performed unless detonation of the substance is observed.

21.4.3.4 *Test criteria and method of assessing results*

21.4.3.4.1 The test results are assessed on the basis of the fragmentation pattern of the tube. The witness plate is used only to provide supplemental information on the violence of the reaction. The test giving the most severe assessment should be used for classification.

21.4.3.4.2 *The test criteria are as follows*:

"Yes" : - the tube is fragmented over its entire length.

"Partial": - the tube is not fragmented over its entire length but the average tube fragmentation (average over the two tests) is greater than 20 cm. (This corresponds to about 1.5 times the fragmentation length found with common inert materials).

"No" : - the tube is not fragmented over its entire length but the average tube fragmentation (average over the two tests) is equal to or less than 20 cm.

21.4.3.5 *Examples of results*

Substance	**Apparent density** (kg/m³)	**Fragmented length** (cm)		**Result**
tert-Butyl peroxyacetate, 75% in solution	-	14		No
tert-Butyl peroxybenzoate	-	16		No
tert-Butyl peroxy-2-ethylhexanoate	-	16		No
Cumyl hydroperoxide, 78% in cumene	-	13		No
Dibenzoyl peroxide	580	40		Yes
Di-tert-butyl peroxide	-	17		No
Di-sec-butyl peroxydicarbonate a/	-	17		No
Diisopropyl peroxydicarbonate a/	1080	40		Yes
2,5-Dimethyl-2,5-di-(tert-butyl-peroxy) hexyne-3	-	21		Partial
2,5-Dimethyl-2,5-di-(benzoylperoxy)-hexane	410		14	No
Di-n-propyl peroxydicarbonate a/	-	40		Yes
Inert substance: Sodium chloride		13		-

a/ Performed at the control temperature"

Replace the diagram.

Replace "21.4.5 Test A.5: UN GAP TEST" with "21.4.4 Test A.6: UN DETONATION TEST".

Renumber all subsequent paragraphs containing 21.4.5.... to 21.4.4....

In old 21.4.5.1, amend the final sentence to read: "... question in box 1 of figure 20.1.".

In old 21.4.5.2, replace the last sentence by:

> "Additional information on the explosive behaviour of the test sample can be gained by the use of a witness plate, as shown in figure 21.4.4.1, or by a velocity probe to measure the velocity of propagation in the substance. The mild steel witness plate of 150 mm square and 3.2 mm thick is mounted at the upper end of the tube and separated from it by spacers 1.6 mm thick."

Delete "21.4.5.3.1 ... figure 20.1)" and the text in square brackets.

Delete old paragraph 21.4.5.3.2.

Delete "21.4.5.5.1 ... figure 20.1)" and the entry in 21.4.5.5

Delete the whole of sub-paragraph 21.4.4.5.2.

Rename the legend of Figure 21.4.5.1 to "UN DETONATION TEST", and replace figure (witness plate to be added, velocity probe and bubbles to be deleted and correct keys A and B accordingly).

Section 22

In 22.4.1.5, in the heading of the table, insert "Apparent" before "Density", and in footnote a/ delete the final sentence.

Section 23

In 23.2.2, insert "is" after "23.2.2 The answer".

In 23.4.1.1, amend "under high confinement" to " under confinement", and add

"When testing liquids, variable results may be obtained because the substance may give two pressure peaks."

In 23.4.1.2.1, delete "copper" in the seventh line.

Amend footnoote to read: "... appendix 4).".

In 23.4.2.1, delete the text in square brackets.

Section 24

Delete 24.2.3.

Section 25

In Table 25.1, delete entry E.5, add to the Dutch pressure vessel test "**/" and add the following legends to the Table:

"*/ Recommended test for self-reactive substances

**/ Recommended test for organic peroxides"

In 25.2.2, replace the text in square brackets by:

"The combination of the Koenen test and either the Dutch pressure vessel or United States pressure vessel test should be used for self-reactive substances. The combination of the Dutch pressure vessel test and either the Koenen test or the United States pressure vessel test should be used for organic peroxides."

In the whole of 25.4, including the legend to the figure, where not stated, amend the orifice plate dimensions with ".0".

In 25.4.1.5, add the following example:

"2,5-Dimethyl-2,5-di(tert-butylperoxy)hexane 23 g 1.5 mm "F" Medium"

In 25.4.1.2.2, amend "3.6" to read "3.3".

In 25.4.1.3.3, replace the text in square brackets with:
"The time to reaction and duration of reaction can provide additional information useful in interpreting the results.".

In 25.4.2.2.1, the first line, amend "24" to "8".

In 25.4.2.2.2, amend "3.7" to read "3.5".

In 25.4.2.3.2, replace the text in square brackets with:

"The time to reaction and duration of reaction can provide additional information useful in interpreting the results.".

In 25.4.2.3.3 and 25.4.2.4.2, replace all references to: "explosion" with "rupture of the disc".

In 25.4.2.4.1, delete the final sentence.

In 25.4.3.2 sub-section (e), replace "670 ± 50" with "620 ± 60".

In 25.4.3.2 sub-section (f), insert in the list of orifice diameters in the right numeric sequence:

"1.2, 3.0, 5.0, 8.0".

In 25.4.3.3.1, delete the last sentence.

In 25.4.3.3.3, replace the last two sentences by:

"In the event of a rupture of the disc the experiment is repeated at the next higher diameter of the orifice until the level is found at which there are no ruptures in three successive experiments."

In 25.4.3.4.2, replace "9 to 24" with "9.0 to 24.0".

In 25.4.3.5, add a footnote to: "4.5" and "18" which reads:

"intermediate diameters, no longer used."

Delete the whole of 25.4.4.

Section 26

In 26.4.3.3.3 replace the square brackets in the formula with "{ }".

In the legend to figure 26.4.3.2, replace "(L) Knurled screw" with "(L) Tin foil" and "(K) Tin foil" with "(K) Knurled screw".

Section 27

Delete 27.2.3.

In table 27.4.2.5, replace "Plastics lined paper bag/carton outer" with "4G".

Section 28

See paragraph 22.

Section 30

No amendments.

Section 31

No amendments.

Section 32

In 32.2.2 (first sentence) insert "2" after "Chapter".

Section 33

In 33.2.1.4.5, insert a second column headed "Particle size" to the table.

In 33.3.1.3.3, incorporate the flowchart given in R.385/Rev.1. Change in the relevant flow chart boxes "$T_{max > 200}$" into "T_{oven} + 60 °C".

In 33.3.1.5.2, first sentence amend "No. 3 Whatman" to read "[small pore size]".

In 33.3.1.5.3.3, first sentence, delete "[No. 3 Whatman]".

In 33.3.1.5.5, insert data supplied by CEFIC to read:

"

Substance	Effect of exposure to air	Effect on filter paper	Result
Di-ethyl aluminium chloride/isopentane (10/90)	No ignition	No charring	Not 4.2
Di-ethyl aluminium chloride/isopentane (15/85)	No ignition	Charring	4.2
Di-ethyl aluminium chloride/isopentane (95/5)	No ignition	Charring	4.2
Tri-ethyl aluminium/heptane (10/90)	No ignition	No charring	Not 4.2
Tri-ethyl aluminium/heptane (15/85)	No ignition	Charring	4.2
Tri-ethyl aluminium/heptane (95/5)	No ignition	Charring	4.2

"

Amend the text for test N.4 (further editorioal amendments are required) as follows:

33.3.1.3.3 Self-heating substances

33.3.1.3.3.1 .....If self-heating occurs with the substance in a 100 mm sample cube then a test with the substance in a 25 mm cube should be performed to determine the packing group. [When no self-heating occurs in a 25 mm sample cube additional test in a 100 mm sample cube at temperatures of 120 °C respectively 100 °C should be performed to determine whether the substance could be considered for exemption when transported in certain packages.] The recommended test method .....

33.3.1.6.2 Apparatus and materials

The following apparatus is required:

a hot-air circulating type of oven with an inner volume of more than 9 litres and capable of controlling the internal temperature at 140+/-2 °C [, 120+/-2 °C and 100+/-2 °C];

33.3.1.6.3 Procedure

[On a negative test result with a 25 mm sample cube additional tests with a 100 mm sample cube should be performed at temperatures of 120 °C respectively 100 °C to determine whether the substance can be considered for exemption when transprted in packages up to 3 m(3) respectively 450 l. A positive test result for both cases is obtained, if spontaneous ignition occurs or if the temperature rise of the sample exceeds 60 K within 24 hours. The principles of classification and packing group assignment of self-heating substances are described in figure xxx.]

33.3.1.6.4 Test criteria and method of assessing results

33.3.1.6.4.1 A substance should be classified in Division 4.2 if in [any of] the test[s] using a 100 mm cube sample, spontaneous ignition occurs or the temperature of the sample exceeds 200 °C [respectively 180 °C or 160 °C] during the 24 hour testing time.

.....

33.3.1.6.4.3 Packing Group III should be assigned to self-heating substances which give positive results when tested with the 100 mm cube sample [at temperatures of 140 °C, 120 °C and 100 °C] but which give a negativ result with a 25 mm cube sample.

[33.3.1.6.4.4 Substances which give a negative test result when tested with a 100 mm sample cube at a temperature of 120 °C and which give a negative result with a 25 mm cube sample are exempted from Division 4.2 when transported in packages with a volume of <= 3 m(3).]

33.3.1.6.4.5 Substances which give a negative test result when tested with a 100 mm sample cube at a temperature of 100 °C and which give a negative result with a 25 mm cube sample are exempted from Division 4.2 when transported in packages with a volume of <= 450l.]

Figure xxx: see document -/C.3/R.385/Rev.1.

Just change the T(max)-values to 180 °C respectively 160 °C

Section 34

34.3.1 add at the end of this paragraph the sentence "As the particle size has a significant effect on the result, the particle size of the substance tested should be stated in the report".

(check whether changes in Appendix 3 are necessary)

Section 35

No amendments.

Section 36

No amendments.

Section 37

No amendments.

Section 38

(other consequential amendments may be required)

In paragraph 38.3.2.3, replace the first sentence by:

"Lithium cells and batteries should be subjected to Series T tests, where applicable, as required by special provisions 188 and 230 prior to the transport of a particular cell or battery type."

In the second sentence, amend "these classification procedures" to read "the required tests".

Amend the first line of paragraph 38.3.4.7.1 to read:

"38.3.4.7.1 A lithium cell or battery, subject to the mass requirements given in special provision 188, is of the type not subject to the Recommendations if:"

Amend the first line of paragraph 38.3.4.7.2 to read:

"38.3.4.7.1 A lithium cell or battery, subject to the mass requirements given in special provision 230, is assigned to Class 9 if:"

Annex E

AMENDMENTS TO THE RECOMMENDATIONS

Chapter 5

Amend paragraph 5.4 by replacing the French standards with:

"France (Association française de normalisation, AFNOR, Tour Europe, Cedex 7, 92080 Paris La Défense):

French Standard NF M 07 - 019
French Standards NF M 07 - 011 / NFT 30 - 050 / NFT 66 - 009
French Standard NF M 07 - 036"

and inserting the Netherlands references:

"Netherlands:

ASTM D93-90
ASTM D3278-89
ISO 1516
ISO 1523
ISO 3679
ISO 3680"

Chapter 11

Insert, after 11.3.13.1, a note reading:

"***Note:*** *An example of a method to determine the size of emergency relief-devices for tank-containers allowed for organic peroxides, as required in 12.550, is given in document ST/SG/AC.10/C.3/R.572 from the European Chemical Industry Council (CEFIC).*"

11.3.4.1 amend "in case of spillage" to read as "in case of spillage or fire"

11.3.4.2 replace last two sentences by "Type B diluents may be used for desensitization of all organic peroxides provided that the boiling point is at least 60 °C higher than the SADT in a 50 kg package".

Chapter 14

In paragraphs 14.2.1.1 and 14.2.1.3.1, insert after "in 14.5.2" the test manual **reference** "(and in sub-section 33.2.1 of the Recommendations: Tests and **Criteria)".**

Add to Table 14.1:

SELF-REACTIVE SUBSTANCE	Concentration (%)	Packing method	Control temperature (°C)	Emergency temperature (°C)	UN generic entry	Remarks
DIETHYLENE GLYCOL BIS(ALLYL-CARBONATE) + DI-ISOPROPYL-PEROXYDICARBONATE	≥88 + ≤12	OP8A	-10	0	3237	

In paragraph 14.2.2.3.3, amend "Part III" to read "Part II".

In paragraph 14.2.2.4.3, delete "for organic peroxides," and insert, after "given in", "Part II of".

As agreed at the eighth session of the Sub-Committee, replace figure 14.2 by the figure (renumbered 14.2 (a) and 14.2 (b)) given in the annex to United Kingdom document ST/SG/AC.10/C.3/R.437. The references to organic peroxides should be deleted.

At the end of paragraphs 14.3.2.1 and 14.3.2.2 append the test manual reference "(see also sub-section 33.3.1 of the Recommendations: Tests and Criteria)".

In paragraphs 14.4.1.1 and 14.4.2.1, insert after "14.5.6" the text "see also sub-section 33.4.1 of the Recommendations: Tests and Criteria)".

Amend 14.5.1.2 to read:
"The tests method and criteria for Class 4 are also given, with advice on application of the tests, in Section 33 of the Recommendations: Tests and Criteria. Test methods and criteria for self-reactive substances are given in Part II of the Recommendations: Tests and Criteria."

In paragraph 14.5.2 (e), add a footnote after "The first test" to read:

"*/ If it is expected that a positive result will be obtained using a 25 mm cube sample then, for safety and environmental protection, the first test may be performed with a 25 mm cube sample. If a positive result is obtained then a test with a 100 mm cube sample is not necessary."